FORM PTO-1390 (REV. 11-2000) U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE ATTORNEY'S DOCKET NUMBER TRANSMITTAL LETTER TO THE UNITED STATES Mo-6284/LeA 32,990 US APPLICATION NO (If known, see 37 CFR 15 DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371 INTERNATIONAL APPLICATION NO. INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED 12 October 1999 (12.10.99) 22 October 1998 (22.10.98) PCT/EP99/07654 TITLE OF INVENTION PHOSGENE HAVING LOW CONTENT OF CARBON TETRACHLORIDE APPLICANT(S) FOR DO/EO/US 1) Jurgen Heuser; 2) Hermann Kauth; 3) Christian Kords Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information: 1. X This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. X The US has been elected by the expiration of 19 months from the priority date (Article 31). 5. X A copy of the International Application as filed (35 U.S.C. 371(c)(2)) is attached hereto (required only if not communicated by the International Bureau). has been communicated by the International Bureau. is not required, as the application was filed in the United States Receiving Office (RO/US). 6. X An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). is attached hereto. has been previously submitted under 35 U.S.C. 154(d)(4). 7. Amendments to the claims of the International Aplication under PCT Article 19 (35 U.S.C. 371(c)(3)) are attached hereto (required only if not communicated by the International Bureau). have been communicated by the International Bureau. have not been made; however, the time limit for making such amendments has NOT expired. have not been made and will not be made. An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)). 9. X An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). Items 11 to 20 below concern document(s) or information included: 11. An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. X An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. X A FIRST preliminary amendment. 14. A SECOND or SUBSEQUENT preliminary amendment. 15. A substitute specification. A change of power of attorney and/or address letter. 16. A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 17. 18. X A second copy of the published international application under 35 U.S.C. 154(d)(4). 19.□ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4), 20. X Other items or information: Preliminary Amendment w/Abstract; PTO Form 1449 and references listed therein

532 Rec'd PET (TO 1-9 APR 2001 INTERNATIONAL APPLICATION NO PCT/EP99/07654 Mo-6284/LeA 32,990 CALCULATIONS PTO USE ONLY The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a (2)) paid to USPTO and International Search Report not prepared by the EPO or JPO International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$860.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO\$710.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT = 860.00 Surcharge of \$130.00 for furnishing the oath or declaration later than \$ months from the earliest claimed priority date (37 CFR 1.492(e)). **CLAIMS** NUMBER FILED NUMBER EXTRA RATE \$ Total claims -20 =x \$18.00 \$ 0.00 Independent claims \$ x \$80.00 3 -3 =0.00 MULTIPLE DEPENDENT CLAIM(S) (if applicable) \$ + \$270.00 0.00 \$ TOTAL OF ABOVE CALCULATIONS =860.00 Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above \$ are reduced by 1/2. 0.00 **SUBTOTAL** \$ 860.00 Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)). \$ TOTAL NATIONAL FEE 860.00 Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be \$ accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property 40.00 TOTAL FEES ENCLOSED 900.00 Amount to be \$ refunded: \$ charged: a. A check in the amount of \$ ______ to cover the above fees is enclosed. Please charge my Deposit Account No. 13-3848 in the amount of \$ 900.00 to cover the above fees. A duplicate copy of this sheet is enclosed. c. X The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3848 ... A duplicate copy of this sheet is enclosed. Fees are to be charged to a credit card. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038. NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status. SEND ALL CORRESPONDENCE TO Aron Preis SIGNATURE

Bayer Corporation Aron Preis Patent Department NAME 100 Bayer Road Pittsburgh, PA 15205-9741 29,426 **USA** REGISTRATION NUMBER

PATENT APPLICATION Mo6284 LeA 32,990

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION	ON OF)
JURGEN HE	EUSER ET AL)
SERIAL NUI	MBER: TO BE ASSIGNED)
FILED:	HEREWITH)
TITLE:	PHOSGENE HAVING LOW CONTENT OF CARBON TETRACHI ORIDE)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents Washington, D.C. 20231 Sir:

Prior to its examination kindly amend the English language translation of the enclosed application as follows:

"Express Mail" mailing label number	EK633383352US
Date of Deposit	Aril 19, 2001
I hereby certify that this paper or fee is	s being deposited with the Office States
1.10 on the date indicated above and is	ce to Addressee" service under 37 CFR addressed to the Assistant Commissioner
1.10 on the date indicated above and is of Patents and Trademarks, Washington	ce to Addressee" service under 37 CFR addressed to the Assistant Commissioner

IN THE SPECIFICATION:

Delete the title of the application, appearing on page 1, and insert therefor:
--Phosgene Having Low content of Carbon Tetrachloride--.

IN THE CLAIMS:

- 4. (Amended) A process according to Claim 2 characterized in that the gas steam emerging from the reactor is under a pressure of 300 kPa_{abs} at most.
- 5. (Amended) A process according to Claim 2 characterized in that the methane content of the carbon monoxide is 50 ppm at most.

Cancel Claim 6.

Add the following:

- --7. A method of using the phosgene of Claim 1 in a process for making a member selected from the group consisting of polycarbonate and polyester carbonate.
- 8. In the process for producing phosgene by reacting carbon monoxide with chlorine the improvement comprising carrying out the reaction in the presence of elemental carbon, restricting the gas stream emerging from said reactor to a temperature of 30 to 80°C to a pressure of 120 to 400 kPa_{abs} as measured directly downstream from said reactor, said phosgene characterized in having a content of carbon tetrachloride that is less than 150 ppm.--

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REMARKS

The present amendment seeks to place the application in better conformance with U.S. practice. A page containing an Abstract of the Disclosure is enclosed.

Entry of the amendment is requested.

Respectfully submitted,

Ву __

Aron Preis

Attorney for Applicants

Reg. No. 29,426

Bayer Corporation 100 Bayer Road Pittsburgh, Pennsylvania 15205-9741 (412) 777-8343 FACSIMILE PHONE NUMBER: (412) 777-8363 /vjt/AP4217

VERSION WITH MARKINGS SHOWING THE CHANGES

IN THE SPECIFICATION:

Please amend the title as follows:

Phosgene which is <u>Having</u> Low in Content of Carbon Tetrachloride.

IN THE CLAIMS:

- 4. (Amended) A process according to elaims 2 or 3, Claim 2 characteriszed in that the gas stream emerging from the reactor is under a pressure of 300 kPa_{abs} at most.
- 5. (Amended) A process according to any one of claims 2 or 4, Claim 2 characteriszed in that the methane content of the carbon monoxide is 50 ppm at most.

Cancel Claim 6.

Add the following Claims:

- --7. A method of using the phosgene of Claim 1 in a process for making a member selected from the group consisting of polycarbonate and polyester carbonate.
- 8. In the process for producing phosgene by reacting carbon monoxide with chlorine the improvement comprising carrying out the reaction in the presence of elemental carbon, restricting the gas stream emerging from said reactor to a temperature of 30 to 80°C to a pressure of 120 to 400 kPa_{abs} as measured directly downstream from said reactor, said phosgene characterized in having a content of carbon tetrachloride that is less than 150 ppm.--

Mo6284 -4-

PCT

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INTERNATIONALE ANMELDUNG VERÖFFENTLICHT NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT)

(51) Internationale Patentklassifikation 7:

C01B 31/28, C08G 64/22, 63/64

(11) Internationale Veröffentlichungsnummer: WO 00/24672

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198 48 668.5

22. Oktober 1998 (22.10.98) DE

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(81) Bestimmungsstaaten: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO Patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), eurasisches Patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI Patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Veröffentlicht

Mit internationalem Recherchenbericht.

(54) Title: PHOSGENE WITH POOR CARBON TETRACHLORIDE CONTENT

(54) Bezeichnung: TETRACHLORKOHLENSTOFFARMES PHOSGEN

(57) Abstract

The invention relates to a method for the production of phosgene having a carbon tetrachloride content of less than 150 ppm, especially for the production of polycarbonates and polyesters. According to said method, carbon monoxide is reacted with chlorine in the presence of elementary carbon at a temperature ranging from 30 to 80° C and a pressure raging from 120 to 400 kPa, said temperature and pressure being measured directly behind the phosgene generator.

(57) Zusammenfassung

In einem Verfahren zur Herstellung von Phosgen mit weniger als 150 ppm Tetrachlorkohlenstoff, insbesondere für die Herstellung von Polycarbonaten und Polyestern, wird Kohlenmonoxid mit Chlor in Gegenwart von elementarem Kohlenstoff bei einer Temperatur von 30 bis 80 °C und einem Druck von 120 bis 400 kPa, gemessen unmittelbar hinter dem Phosgengenerator, umgesetzt.

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Phosgene which is low in carbon tetrachloride

This invention relates to phospene which is low in carbon tetrachloride, to a process for the production thereof, and to the use thereof for producing polycarbonates and polyester carbonates.

Phosgene is an important starting material for producing intermediates and final products in many branches of chemistry, particularly for the production of isocyanates, polycarbonate plastics and esters of chloroformic acid. Pure phosgene is a colourless, toxic gas which is susceptible to hydrolysis. Under normal pressure it boils at -127.8°C and condenses at 7.56°C to form a colourless liquid.

It is known that phosgene can be produced by the catalytic combination of carbon monoxide (CO) and elemental chlorine (Cl₂):

$$CO + Cl_2 \xrightarrow{Catalysis} C=O$$

$$CI$$

$$C = O$$

$$C = O$$

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Activated carbon is mostly used as the catalyst. The reaction is exothermic and the conversion is almost complete. The reaction is usually conducted in tubular reactors which are made from standard steel or stainless steel and which are packed with catalyst. These tubes are generally cooled externally in order to dissipate the heat of reaction. Depending on the processing conditions, the phosgene which is produced by this process contains varying amounts of carbon tetrachloride (CCl₄) as an impurity. This is formed by unwanted secondary reactions such as disproportionation reactions, excessive chlorination of the carbon monoxide, chlorination of the carbon catalyst and chlorination of methane (CH₄). The degree of contamination by carbon tetrachloride of phosgene which is produced by commonly-used processes usually falls within the range from 250 to 2000 ppm.

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April 19, 2001
is being deposited with the United States fice to Addressee" service under 37 CFR addressed to the Assistant Commissioner n, D.C. 20231
a J. Veatch malling paper or fee)
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The formation of carbon tetrachloride as a by-product during the production of phosgene is problematical from many aspects. Thus, due to the environmental harmfulness of chlorinated hydrocarbons, costly precautions have to be taken to ensure that the carbon tetrachloride formed in the reactions is not discharged into the environment. Moreover, even slight contents of carbon tetrachloride have a disadvantageous effect on some products which are manufactured from phosgene, particularly plastics. For example, high-purity phosgene with a carbon tetrachloride content of 250 ppm at most is required for the production of polycarbonate plastics, in order to prevent discoloration of the final product, which is normally colourless and transparent.

JP 88-156040 describes a process for producing phosgene which is low in carbon tetrachloride by the reaction of carbon monoxide with chlorine over activated carbon at 350°C, wherein the activated carbon catalyst is purified before reaction by treatment with acid to remove impurities such as transition metals, boron, aluminium and silicon. The phosgene which is produced by this process has a carbon tetrachloride content of 150 ppm. A disadvantage of this process, however, is the costly pretreatment of the activated carbon catalyst.

JP 80-014044 proposes a reactor for the conversion of chlorine and carbon monoxide into phosgene, in which the activated carbon catalyst is positioned at the reactor walls with the aid of a metal mesh. This arrangement prevents overheating of the catalyst bed. The phosgene which is produced in a reactor such as this at temperatures of 200 to 250°C and at a throughput of 600 to 1200 l/h has a carbon tetrachloride content of 250 to 400 ppm. A disadvantage of this process, however, is that repacking the reactor with activated carbon when the activity of the latter decreases is more expensive than is the operation of conventional tubular reactors.

The underlying object of the present invention is to provide phosgene with an even lower carbon tetrachloride content.

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This object is achieved by a grade of phosgene which contains less than the 150 ppm of carbon tetrachloride. The present invention further relates to a process for producing a grade of phosgene such as this by the reaction of carbon monoxide with chlorine in the presence of elemental carbon at a temperature of 30 to 80°C and at a pressure of 120 to 400 kPa_{abs} as measured directly downstream of the phosgene generator.

It is advantageously possible to conduct the process according to the invention in customary tubular reactors made of standard steel or stainless steel, the tubes of which are packed with the carbon catalyst. The tubular reactor can be operated continuously or batch-wise.

Carbon tetrachloride and chlorine are introduced into the reactor in approximately equal parts, preferably at room temperature. To ensure that all the chlorine is reacted, a slight excess of carbon monoxide can be used. Before they enter the reactor, the two reactants are preferably mixed in a suitable mixing device, in a static mixer for example.

Another advantage of the process according to the invention is that no special preparation of the catalyst is necessary.

The temperature of the gas stream emerging from the reactor should not exceed 70 to 80°C, as measured directly downstream of phosgene generator. The temperature of the gas stream emerging from the reactor in the process according to the invention is preferably 40 to 70°C. The gas stream is prevented from exceeding these temperatures with the aid of appropriate cooling devices which dissipate the heat of reaction which is released during the reaction and which prevent any overheating of the catalyst.

Activated carbon is preferably used as the carbon catalyst. Granular activated carbon with a grain diameter of 3 to 4 mm is preferably used as the catalyst. The pore surface area of the activated carbon is preferably about 1000 m²/g. The apparent density of the activated carbon used is preferably about 450 g/l.

The pressure which is measured directly downstream of the phosgene reactor is preferably 300 kPa_{abs}, at most. It is thereby ensured that phosgene cannot condense in the reactor.

The phosgene which is produced by the process according to the invention and which emerges from the top of the reactor is preferably condensed at temperatures of -10 to -20°C. Due to its low carbon tetrachloride content of less than 150 ppm, it can be used directly, without further purification, for producing polymers comprising a carbonate bond, particularly polycarbonates or polyester carbonates.

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The chlorine which is used in the reaction can be produced by customary industrial processes such as chlor-alkali electrolysis or hydrogen chloride electrolysis, and should be as pure as possible. Chlorine with a degree of purity higher than 98 % is particularly suitable. Liquid chlorine from a storage vessel is preferably used. This is evaporated in a heated gasifier and is subsequently freed from any entrained liquid chlorine in a reevaporator.

The carbon monoxide which is used in the reaction can be produced by customary methods, for example from natural gas/naphtha in a synthesis gas installation or by blowing coke with oxygen. It has proved to be particularly advantageous if the carbon monoxide has a methane content of less than 50 ppm.

The invention is explained in more detail below by means of examples.

- 5 -

Example 1

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500 Nm³/h carbon monoxide with a purity of 97.5 % and a methane content of 50 ppm as determined by on-line IR measurement (Method 2301-0207901-91D), and 480 Nm³/h chlorine with a purity of 99.9 % were mixed at room temperature and were fed to a phosgene generator packed with activated carbon (grain diameter about 4 mm, pore surface area about 1000 m²/g, apparent density about 450 g/l). The generator was cooled so that the temperature of the phosgene formed, as measured directly after the phosgene left the generator, was 55°C. The pressure as measured directly after the phosgene left the generator was 80 kPa_{gauge} (180 kPa_{abs}). The phosgene produced had a carbon tetrachloride content of 100 ppm.

Example 2

500 Nm³/h carbon monoxide with a purity of 98 % and a methane content of 50 ppm as determined by on-line IR measurement (Method 2301-0207901-91D), and 480 Nm³/h chlorine were mixed at room temperature and were fed to a phosgene generator packed with activated carbon (grain diameter about 4 mm, pore surface area about 1000 m²/g, apparent density about 450 g/l). The generator was cooled so that the temperature of the phosgene formed, as measured directly after the phosgene left the generator, was 40°C. The pressure as measured directly after the phosgene left the generator was 40 kPa_{gauge} (140 kPa_{abs}). The phosgene produced had a carbon tetrachloride content of 80 ppm.

Comparative example

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500 Nm³/h carbon monoxide with a purity of 98% and a methane content of 10 ppm were mixed at room temperature with 485 Nm³/h chlorine and were fed to a phosgene generator as in Examples 1 and 2. The generator was cooled so that the temperature of the phosgene formed, as measured directly after it left the generator, was 85°C. The pressure, which was likewise measured directly after the phosgene left the generator, was 600 kPa_{gauge} (700 kPa_{abs}). The phosgene produced had a carbon tetrachloride content of 290 ppm.

Claims

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- 1. Phosgene which is low in carbon tetrachloride, containing less than 150 ppm of carbon tetrachloride.
- A process for producing phosgene which is low in carbon tetrachloride by the reaction of carbon monoxide with chlorine in the presence of elemental carbon in a reactor, characterised in that the gas stream emerging from the reactor is at a temperature of 30 to 80°C and is under a pressure of 120 to 400 kPa_{abs} as measured directly downstream of the phosgene generator.
 - 3. A process according to claim 2, characterised in that the gas stream emerging from the reactor is at a temperature of 40 to 70°C.
- A process according to claims 2 or 3, characterised in that the gas stream emerging from the reactor is under a pressure of 300 kPa_{abs} at most.
 - 5. A process according to any one of claims 2 to 4, characterised in that the . methane content of the carbon monoxide is 50 ppm at most.
 - 6. Use of the phosgene which is low in carbon tetrachloride according to any one of claims 1 to 5 for producing polycarbonates and polyester carbonates.

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PCT/EP99/07654 532 Rec': 19 APR 2001

PHOSGENE HAVING LOW CONTENT OF CARBON TETRACHLORIDE

ABSTRACT OF THE DISCLOSURE

A process for producing phosgene having low content of carbon tetrachloride is disclosed. The process entails reacting carbon monoxide with chlorine in the presence of elemental carbon, in a reactor. Critically the temperature of the gas stream emerging from the reactor is restricted to 30 to 80°C and its pressure, measured directly downstream from said reactor is restricted to 120 to 400 kPa_{abs}. The phosgene thus produced has a content of carbon tetrachloride that is less than 150 ppm.

COMBINED DECLARATION AND POWER OF ATTORNEY

ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought

on the invention entitled

"PHOSGENE WITH POOR CARBON TETRACHLORIDE CONTENT"

the specification of which is attached hereto,

or was filed on October 12, 1999

as a PCT Application Serial No. PCT/EP99/07654

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, \$119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

198 48 668.5 (Number)

Germany (Country)

Otober 22, 1998 (Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, \$120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, \$112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, \$1.56 which occured between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status)
		(patented, pending, abandoned)
(A 1: .: G : 1 3T)	(Dili D-+-)	(Status)
(Application Serial No.)	(Filing Date)	(Status)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

JOSEPH C. GIL, Patent Office Registration Number 26,602
ARON PREIS, Patent Office Registration Number 29,426
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THOMAS W. ROY, Patent Office Registration Number 29,582
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	FULL NAME OF SIXTH INVENTOR	INVENTOR'S SIGNATURE		DATE		
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	FULL NAME OF SEVENTH INVENTOR	INVENTOR'S SIGNATURE		DATE		
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